

Standard range: 0.01 to 1 mg/L Fe, Total

Method

EZ2724



Test preparation

Before starting

Chemical exposure hazard. Obey laboratory safety procedures and wear all of the personal protective equipment appropriate to the chemicals that are handled. Refer to the current safety data sheets (MSDS/SDS) for safety protocols.

Review the Safety Data Sheets (MSDS/SDS) for the chemicals that are used. Use the recommended personal protective equipment. Dispose of chemicals and wastes in accordance with local, regional and national regulations.

Review the Safety Data Sheets (MSDS/SDS) before the bottles are filled or the reagents are prepared.

All chemicals must be of reagent grade, ACS grade or better¹. The use of pro-analysis chemicals is recommended. Use of reagents that are not of sufficient quality can have a negative effect on the analyzer performance.

All EZ analyzers are put through long tests with standard solutions, reagents and dilution water prepared with Type I water or better water as specified in ASTM D1193-91.

To get the specifications shown on the data sheet, method and reagents sheet and acceptance test reports, the same water quality (or better) must be used to prepare the standard solutions, reagents and dilution water.

In addition, prepare the standard solutions for an EZ analyzer with water that does not contain the parameter to be measured or interferences for the method.

When operating the device, always make sure to follow the reagent recommendations given in [Reagent consumption](#) on page 3.

For longer-term storage, keep the reagents in a cold and dark place. Do not keep reagents longer than recommended. If applicable, keep the reagents in a refrigerator during measurements. Refer to [Reagent consumption](#) on page 3 for the reagent temperature.

The manufacturer recommends to replace the reagents, stock and standard solution at 28-day intervals unless specified differently in the sections that follow. Do not mix used reagents with freshly prepared reagents. If reagents, standards or DI water in the containers are replaced, discard all of the container contents in accordance with local, regional and national regulations. Rinse out all of the containers and then fill each container with freshly prepared new reagent.

Specifications

Specifications are subject to change without notice.

| Specification | Details |
|------------------------|--|
| Analysis method | TPTZ colorimetric method |
| Measurement wavelength | 578 nm |
| Parameter | Long description: Iron, Total Short description (default): Fe, Total Options: None |
| Unit | mg/L (default); ppm, ppb, µg/L |
| Precision | The precision value is found on the full-scale range for standard test solutions. Refer to Table 1 . |
| Cleaning | Automatic or manual; frequency is freely programmable |

¹ Analytical Reagent (AR), Guaranteed Reagent (GR), UNIVAR, AnalalR, Premium Reagent (PR), ReagentCertified ACS reagent, ACS Plus reagent, puriss p.a. ACS reagent, ReagentPlus®, TraceCERT®, Suprapur®, Ultrapur®, or better are also possible.

| Specification | Details |
|---------------|---|
| Calibration | Automatic or manual; 2-point, offset or slope; frequency is freely programmable Note: The manufacturer recommends that a calibration is done when the reagents are replaced. |
| Validation | Automatic or manual; frequency is freely programmable |
| Interferences | Metal ions, e.g., cadmium (Cd), chromium (Cr), cobalt (Co), copper (Cu)], cyanide (CN ⁻), manganese (Mn), mercury (Hg), molybdenum (Mo), nickel (Ni ²⁺) and nitrite (NO ₂ ⁻). Large quantities of color and turbidity interfere. Fats, oil, proteins, surfactants and tar interfere. |

Table 1 Measuring ranges

| Range code | Description | LOD (mg/L) | Range (mg/L) | Precision (%) | Cycle time (minutes) | |
|------------|---|-------------|--------------|---------------|----------------------|-----------|
| | | | | | Continuous | Default |
| B | 25% of standard range | 0.005 | 0.25 | 2 | 20 | 30 |
| C | 50% of standard range | 0.005 | 0.5 | 2 | 20 | 30 |
| 0 | Standard range | 0.01 | 1 | 2 | 20 | 30 |
| V | internal dispenser dilution (factor 5) | 0.05 | 5 | 2 | 23 | 30 |
| W | internal dispenser dilution (factor 10) | 0.1 | 10 | 2 | 23 | 30 |
| X | internal dispenser dilution (factor 25) | 0.25 | 25 | 2 | 23 | 30 |

Summary of method

Summary

Ferric iron (Fe³⁺) in the water sample is reduced to ferrous iron (Fe²⁺) by a reducing agent. The ferrous iron then reacts with 2,4,6-tripyridyl-1,3,5-triazine at a pH of 4.5 (± 0.2) to form a blue color. The absorbance is measured at a wavelength of 578 nm. The sample is first digested with heat and the reducing agent to make sure that all forms of iron are measured.

Analysis steps

The analyzer mixes the sample with the reducing agent in the digester vessel and heats the solution to 110 °C (230 °F) for 10 minutes (default digestion temperature and time). The undissolved and complexed forms of iron break apart into a reactive form. After digestion, the sample temperature is decreased until sufficiently cool.

The digested sample is then moved into the analysis vessel. The buffer reagent is added and the initial absorbance value is measured. The color reagent is then added and a stir period starts.

After the stir period, the color is fully developed and the final absorbance value is measured. The analyzer uses the absorbance values and Beer's Law to calculate the concentration of iron in the sample.

Calibration

The calibration procedure measures the REF1 solution (Channel 9, REF1 valve) and the REF2 solution (Channel 10, REF2 valve).

Validation

The validation procedure measures the REF2 solution (Channel 10, REF2 valve).

Reagent consumption

[Table 2](#), [Table 3](#) and [Table 4](#) show the consumption rate of the reagents and calibration standards. Examine the consumption of the reagents after 28 days to adjust the quantities prepared. Refer to [Necessary reagents](#) on page 4 to collect the necessary items to prepare the reagents.

Table 2 Reagent consumption

| Product information | | | Consumption | | | Recommendation | | |
|---------------------|-----------|----------------|---------------|--|----------|---------------------|---------------------------|--|
| Code | Label | Product | Each analysis | Per 28 days, rate of 1 analysis/30 minutes | Use life | Containers | Operation temperature | |
| Red | Reagent 1 | Buffer | ~ 1 mL | ~ 1.4 L | 28 days | Plastic; 2.5 L | 10 to 30 °C (50 to 86 °F) | |
| Blue | Reagent 2 | Color | ~ 0.8 mL | ~ 1.1 L | 28 days | Glass, amber; 2.5 L | 10 to 30 °C (50 to 86 °F) | |
| Green | Reagent 3 | Reducing agent | ~ 0.5 mL | ~ 0.7 L | 28 days | Plastic; 2.5 L | 10 to 30 °C (50 to 86 °F) | |

Table 3 Calibration standards

| Product information | | Consumption | | Recommendation | | |
|---------------------|---------------|-----------------|----------|--|--|--|
| Label | Product | Per calibration | Use life | Containers | | |
| REF1 | REF1 standard | ~ 0.2 L | 28 days | Plastic, 1 L (align with recommendation) | | |
| REF2 | REF2 standard | ~ 0.2 L | 28 days | Plastic, 1 L (align with recommendation) | | |

Table 4 Calibration recommendations

| Calibration | Time (minutes) | | Recommended frequency | Solutions |
|-----------------------|----------------|---------------|-------------------------------|---------------|
| | No dilution | With dilution | | |
| Offset | 61 | 70 | — | REF1 |
| 2-point (recommended) | 123 | 141 | Reagent replacement (28 days) | REF1 and REF2 |

DI water consumption

The volumes shown in [Table 5](#) are an estimation of the consumption for rinse and dilution water based on a standard operating procedure as given in the specifications of the EZ analyzer.

Note: Rinse water volumes can increase because of the sample matrix.

Note: The range codes B, C, 0 are configured as default without the use of rinse and dilution water.

Table 5 DI water consumption

| Range code | Rinse water Type I (mL/analysis) | Dilution water Type I (mL/analysis) | Total (mL/analysis) | Per 28 days, rate of 1 analysis each 30 minutes |
|---------------------------|----------------------------------|-------------------------------------|---------------------|---|
| B - C - 0 (no dilution) | — | — | — | — |
| V - W - X (with dilution) | 63 mL | 16 mL | 79 mL | 107 L |

Rinse water

If the analyzer does a dilution, a deionized water rinse must be used. If no dilution is done, use the sample to rinse. If there is a filter panel in front of the analyzer, make sure that the rinse water also flows through the filter.

Necessary reagents

A reagent kit with mixing instructions is available that decreases the preparation time. Refer to [Table 6](#). The full list of reagents is shown in [Table 7](#). The product name, formula, molecular weight, CAS number and the necessary quantity to prepare 1 L of the reagents are given.

Table 6 Reagent kit for EZ2724: APPC1024KTO

| Code | Label | Product | Quantity | Item no. |
|-------|----------------|----------------|-------------|-------------|
| Red | Reagent 1 | Buffer | 2 L (1x) | APPC1024-01 |
| Blue | Reagent 2 | Color | 2 L (1x) | APPC1024-02 |
| Green | Reagent 3 | Reducing agent | 2 L (1x) | APPC1024-03 |
| — | Stock solution | 10 mg/L Fe | 500 mL (1x) | 14049 |

Table 7 Reagent list

| Solutions | Products | Formula | MW (g/mol) | CAS number | For each 1 L solution |
|--|--|---|------------|------------|---|
| Reagent 1: Buffer Code: Red | Ammonium acetate | C ₂ H ₇ NO ₂ | 77.08 | 631-61-8 | 250 g |
| | Acetic acid | C ₂ H ₄ O ₂ | 60.05 | 64-19-7 | 350 mL |
| Reagent 2: Color Code: Blue | 2,4,6-trypyridyl-1,3,5-triazine | C ₁₈ H ₁₂ N ₆ | 312.33 | 3682-35-7 | 0.750 g |
| | Hydrochloric acid (1 M) | HCl | 34.46 | 7647-01-0 | 100 mL |
| Reagent 3: Reducing reagent Code: Green | Hydroxylamine hydrochloride | H ₃ NO * HCl | 69.49 | 5470-11-1 | 100 g |
| | Hydrochloric acid (1 M) | HCl | 34.46 | 7647-01-0 | 200 mL |
| Stock solution | Ammonium iron (II) sulfate hexahydrate | (NH ₄) ₂ Fe(SO ₄) ₂ * 6H ₂ O | 392.14 | 7783-85-9 | 7.021 g |
| | Sulfuric acid (96%) | H ₂ SO ₄ | 101.19 | 7664-93-9 | 5 mL |
| REF1 calibration standard | Deionized water (Type I or better) | — | — | — | — |
| REF2 calibration standard | 1000 mg/L Fe stock solution | — | — | — | Refer to Table 8 on page 6. |
| | Sulfuric acid (1 M) | H ₂ SO ₄ | 101.19 | 7664-93-9 | Refer to Table 8 on page 6. |
| | Hydroxylamine hydrochloride | H ₃ NO * HCl | 69.49 | 5470-11-1 | Refer to Table 8 on page 6. |
| Validation standard (optional) | REF2 calibration standard | — | — | — | Refer to Validation standard on page 6. |
| Cleaning solution (optional) | Hydrochloric acid (36%) | HCl | 36.46 | 7647-01-0 | 41.5 mL |

Reagent preparation

As an alternative to the reagent kit, the user can prepare the reagents as follows. Refer to [Table 7](#) on page 4 to collect the applicable items. To calculate the correct reagent quantity, refer to [Reagent consumption](#) on page 3.

Make sure to discard the remaining solution from the analyzer bottles before new reagents are added.

Reagent 1: Buffer

1. Add 500 mL of deionized water to a beaker.
2. Add 250 g of ammonium acetate ($\text{C}_2\text{H}_7\text{NO}_2$).
3. Mix until fully dissolved.
4. Add 350 mL of acetic acid ($\text{C}_2\text{H}_4\text{O}_2$).
5. Mix until fully dissolved.
6. Pour the solution into a 1000-mL volumetric flask.
7. Add deionized water to the mark.
8. Fully mix the solution.

The pH value of the buffer reagent should be 4.5 (± 0.2).

Reagent 2: Color

1. Add 500 mL of deionized water to a beaker.
2. Add 0.750 g of TPTZ (2,4,6-tripyridyl-1,3,5-triazine).
3. Mix until fully dissolved.
4. Add 100 mL of 1 M hydrochloric acid solution (HCl).
5. Mix until fully dissolved.
6. Pour the solution into a 1000-mL volumetric flask.
7. Add deionized water to the mark.
8. Fully mix the solution.

Reagent 3: Reducing agent

1. Add 300 mL of deionized water to a beaker.
2. Add 100 g of hydroxylamine hydrochloride ($\text{H}_3\text{NO}^+ \cdot \text{HCl}$).
3. Mix until fully dissolved.
4. Add 200 mL of 1 M hydrochloric acid solution (HCl).
5. Pour the solution into a 1000-mL volumetric flask.
6. Add deionized water to the mark.
7. Fully mix the solution.

Calibration standards

Calibrations are completed with two standards: a REF1 calibration standard and a REF2 calibration standard. The REF2 calibration standard is a dilution of a stock solution.

Stock solution

Prepare a 1000-mg/L Fe stock solution as follows. Refer to [Necessary reagents](#) on page 4 to collect the applicable items.

1. Add 300 mL of deionized water to a beaker.
2. Add 7.021 g ammonium iron (II) sulphate hexahydrate [$(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$].
3. Carefully add 5 mL of concentrated sulfuric acid (H_2SO_4 , 96%).
4. Fully mix the solution.
5. Pour the solution into a 1000-mL volumetric flask.
6. Add deionized water to the mark.
7. Fully mix the solution.

REF1 calibration standard

Use deionized water for the REF1 calibration standard.

REF2 calibration standard

Dilute the stock solution to prepare the REF2 calibration standard.

1. Prepare a hydroxylamine hydrochloride solution as follows:
 - a. Add 500 mL of deionized water to a beaker.
 - b. Add 100 g of hydroxylamine hydrochloride.
 - c. Mix until fully dissolved.
 - d. Pour the solution into a 1000-mL volumetric flask.
 - e. Add deionized water to the mark.
 - f. Fully mix the solution.
2. Use a pipet to add the applicable quantity (mL) of the stock solution into a 1000-mL volumetric flask. Refer to [Table 8](#).
3. Use a pipet to add the applicable quantity (mL) of 1 M H_2SO_4 into the volumetric flask. Refer to [Table 8](#).
4. Use a pipet to add the applicable quantity (mL) of hydroxylamine hydrochloride into the volumetric flask. Refer to [Table 8](#).
5. Add deionized water to the mark.
6. Fully mix the solution.

Table 8 Calibration standard preparation

| Range code | REF2 concentration (mg/L Fe) | Quantity (mL) of stock solution | Quantity (mL) of 1 M H_2SO_4 | Quantity (mL) of hydroxylamine hydrochloride |
|------------|------------------------------|---------------------------------|--|--|
| B | 0.25 | 0.25 | 2.5 | 1 |
| C | 0.5 | 0.5 | 2.5 | 1 |
| 0 | 1 | 1 | 2.5 | 1 |
| V | 5 | 5 | 2.5 | 1 |
| W | 10 | 10 | 2.5 | 1 |
| X | 25 | 25 | 2.5 | 1 |

Validation standard

By default, the automatic validation procedure is not enabled. When enabled, the default validation standard is the REF2 calibration standard. For best results, use a different standard solution from a different source for the validation standard. The concentration of the validation standard must be within the measuring range of the analyzer.

Before validation, connect the REF2 sample line to the validation standard. After validation, connect the REF2 sample line to the REF2 calibration standard again. For multi-channel setups, a different channel can be used.

Cleaning solution

By default, the automatic cleaning procedure is not enabled. When enabled, the default volume of cleaning solution that is used during each cleaning cycle is 30 mL.

The cleaning procedure must prevent the collection of chemicals in the analyzer. For an accurate cleaning procedure, examine the cleaning solution and the cleaning interval for each application. Make sure that the cleaning procedure is sufficient. Change the cleaning procedure if necessary.

The manufacturer recommends to use a 0.5 M hydrochloric acid (HCl) solution. Refer to [Necessary reagents](#) on page 4. Prepare the solution as given in the steps that follow or use a commercially available solution.

1. Add 500 mL of deionized water to a beaker.
2. Slowly mix in 41.5 mL of concentrated hydrochloric acid (HCl, 37%).
3. Pour the solution into a 1000-mL volumetric flask.

4. Add deionized water to the mark.
5. Fully mix the solution.



FOR TECHNICAL ASSISTANCE, PRICE INFORMATION AND ORDERING:
In the U.S.A. – Call toll-free 800-227-4224
Outside the U.S.A. – Contact the HACH office or distributor serving you.
On the Worldwide Web – www.hach.com; E-mail – techhelp@hach.com

HACH COMPANY
WORLD HEADQUARTERS
Telephone: (970) 669-3050
FAX: (970) 669-2932